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**WEAR AND FRICTION
CHARACTERISTICS OF SELF-
LUBRICATING COPPER -
INTERCALATED GRAPHITE COMPOSITES**

**A. W. Ruff, M. B. Peterson,
A. Gangopadhyay, E. Whitenton**

1989

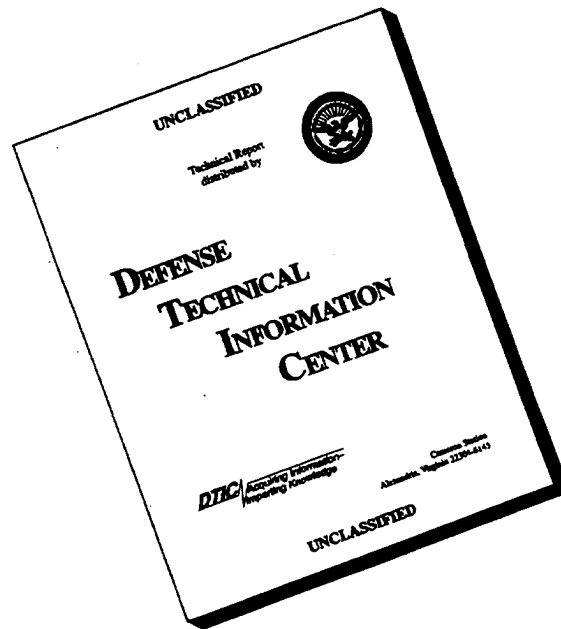
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WEAR AND FRICTION CHARACTERISTICS OF SELF-LUBRICATING COPPER - INTERCALATED GRAPHITE COMPOSITES

A.W. Ruff, M.B. Peterson, A. Gangopadhyay,
and E. Whiteman*

Composite materials consisting of copper metal- matrices with a solid lubricant phase of intercalated (NiCl_2) graphite have been prepared and studied in sliding wear against type 440C stainless steel at normal temperatures in air. Results on the controlling wear and friction mechanisms in these materials are presented. Beneficial effects were found up to about 15 volume percent intercalated graphite. An analytical model has been developed that relates composite wear to mechanical and tribological properties of the different solid phases in the composite and the interface film.

1 INTRODUCTION

Self-lubricating materials offer attractive options for many tribological applications where liquid lubrication is not feasible, where thin solid film lubrication is not durable, and where long, unattended operating life is required. The use of solid lubricants as contrasted to liquid lubricants offers broad application potential¹ including higher temperatures, vacuum environments, sealed systems, and long storage life. The present work is part of a program examining basic issues of tribological and mechanical performance in solid, multi-phase, self-lubricating composites. This report discusses results on a metal-matrix (copper) composite using intercalated (NiCl_2) graphite² (referred to hereafter as IG) as the solid lubricating phase. Previous research³ on metal-matrix and polymer-matrix composites with other solid lubricating phases has shown beneficial effects.

2 EXPERIMENTAL APPROACH

Sliding wear tests were carried out using two configurations of a pin-on-ring tribological test system. One configuration permitted loading two pins simultaneously against a rotating steel ring; the other utilized a holder carrying a single pin (Fig.1). The rings were conventional tapered bearing cups, 35 mm diameter, 8 mm wide, type 440C stainless steel, hardness HRC 58, surface finish 0.15 μm RMS. Tribological tests were carried out in laboratory air at normal ambient temperature. Ambient

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relative humidity ranged from 10% to 40%. The load was 33 N, sliding speed 0.14 m/s, sliding distance usually 790 m. Specimen pins of 6.25 mm diameter were fabricated by several methods. Compaction of powders was done in a hydraulic press using a die at forces of 5,000 to 10,000 lbf (22 to 44 kN). Pure compacts of copper and of IG were made for the 2-pin tests. Compacts of pre-mixed powder combinations of copper and IG were made for the single pin tests. Figure 2 shows an example of the microstructure of such a mixed compact. Two types of manufactured composites were also used: one containing a series of holes in an hexagonal arrangement with different hole diameters and separations, and the other contained a central slit with different widths (Fig. 3). These

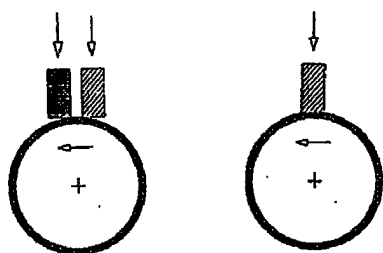


Fig. 1. Schematic of wear geometry using either one or two specimen pins loaded against a rotating ring.



Fig. 2. SEM photograph of Cu-57 vol.% IG powder compacted specimen.

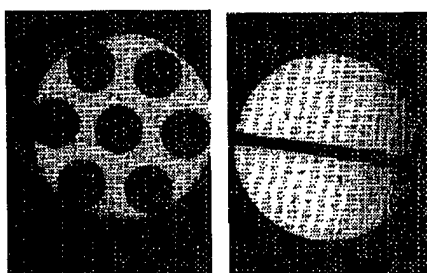


Fig. 3. Optical photographs of manufactured Cu - IG composite specimens. Hole size, hole separation, and slit width were varied.

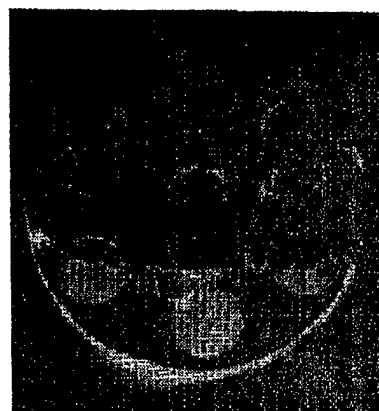


Fig. 4. Wear scar on manufactured Cu - IG composite pin, 6.25 mm diameter.

size variations permitted different effective area and volume percentages of IG and copper under controlled phase shape and spacing.

3 RESULTS AND DISCUSSION

The overall appearance of a typical wear scar on a pin is shown in Fig. 4 for a manufactured composite of Cu-IG. Pin wear volumes were calculated from the pin wear scar dimensions using numeric integration methods. Wear on the rotating ring counterface was not appreciable, although there was some small contribution to the wear debris produced. Figure 5 shows a higher magnification view within the wear scar on a powder composite specimen. Figure 6 shows more detail of the worn surface morphology near the IG phase region in a manufactured composite pin. In both types of specimens, wear led to the establishment of an interfacial film, to the generation of wear debris particles some of which remained in the contact gap, and to surface recession at the location of the soft IG phase. Each of these characteristic features is important in determining the wear and friction mechanisms involved in the tribology of self-lubricating composites.

The interfacial film formed during sliding wear was found on both the composite pin and on the steel ring. While patchy in its distribution and non-uniform in thickness, it is clear that this film is a principle factor controlling wear and friction behavior in this type of system. The interfacial film is comprised of system wear products, namely, IG, copper, and steel (probably Fe, Cr, Ni oxides), based on x-ray microanalysis of the film. Figure 7 shows an SEM photograph from an area on the sliding surface of a manufactured composite pin, near an IG phase region. Sliding is from right to left in this case, and the film being formed on the pin at the exit side of the IG phase region can be clearly seen. X-ray spectra from two areas (marked A and B) are shown. In both spectra,

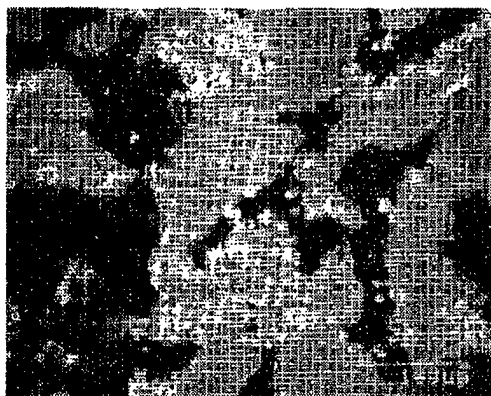


Fig. 5. SEM photograph of worn area in Cu-57 vol.% IG powder compacted specimen.

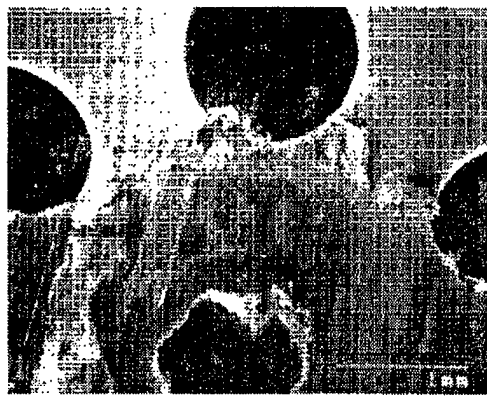


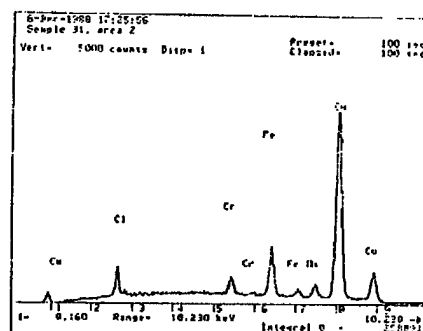
Fig. 6. SEM photograph of worn area on manufactured Cu-25 vol.% IG composite specimen. Sliding direction downward.

the Cu peak originates from wear products in the film, as do the Fe, Cr, and Ni peaks. The Cl peak can be used to determine the amount of IG present (coming from the NiCl_2 intercalation). At thicker regions of the film such as at B, we find proportionally greater amounts of IG (denoted by the Cl peak), and of ring wear debris. Wear debris was not found imbedded into the soft IG phase region. Similar morphology and composition evidence to this has been found in the particulate composite specimens, such as shown in Fig. 5.

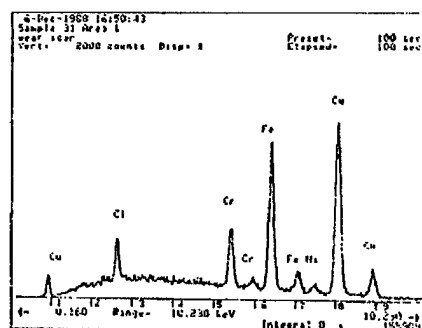
Measured friction and wear data on both types of composites are shown in Figs. 8 and 9. In each case 3 to 5 individual tests were conducted for each of the indicated values of volume % (or area %) IG; average values and a range of \pm one standard deviation are shown. In Fig. 8 the dashed line shows the measured friction value for the 2-pin tests of copper and IG; this result is taken as a baseline for friction since the separate IG pin can supply as much material as needed according to its wear rate. It is seen that friction decreases from a value of about 0.5 for copper to values about 0.15 as the IG fraction in the composite increases. Most of the beneficial decrease occurs up to about 15 vol. % IG. The wear rate for the composites is shown in Fig. 9a. The dashed line again refers to the wear of a Cu pin in 2-pin tests. A reduction in wear occurs on adding up to about 15 vol. % IG. Higher amounts of IG lead to increased wear. This type of behavior suggests that two processes are involved. It is thought that since higher amounts of IG are associated with lower copper content in the composite, that mechanical weakening in the supporting copper matrix takes place at higher levels of IG content and this leads to increased wear. If true, then normalization of the wear data by the



a



b



c

Fig. 7. (a) SEM photograph of worn area in Cu-25 vol.% IG composite. Sliding direction is right to left. Wear debris is collected at entrance to the recessed IG phase region. Interface film formed at exit of IG phase region. (b) X-ray microanalysis from interface film at location A and (c) location B show proportion of IG, Cu, and Fe, Cr, Ni from steel counterface.

vol. % (or area %) Cu in each composite should remove the influence on the wear data of the matrix strength. This is equivalent to applying a rule-of-mixtures for the composite strength influence. Figure 9b shows the normalized wear rate data obtained by this method. It is seen that a monotonic decrease in normalized wear occurs with increasing IG fraction, and that most of the beneficial effect is accomplished by adding up to 15 vol. % IG. This decrease with increasing fraction of IG is consistent with the wear model described next.

4 WEAR MODEL

The steady state thickness of the IG interfacial film is affected by (1) growth as IG is worn away from the composite and added to the film, and (2) shrinkage as the film itself is worn away. Therefore

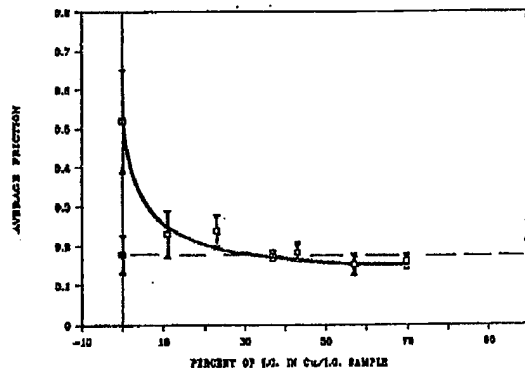
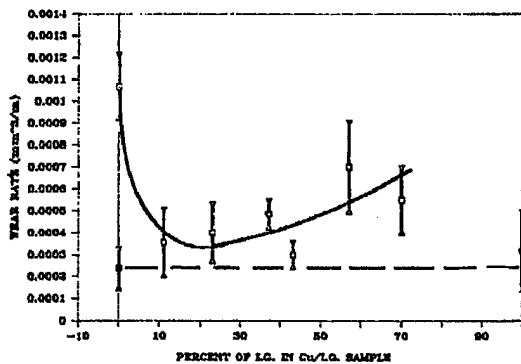
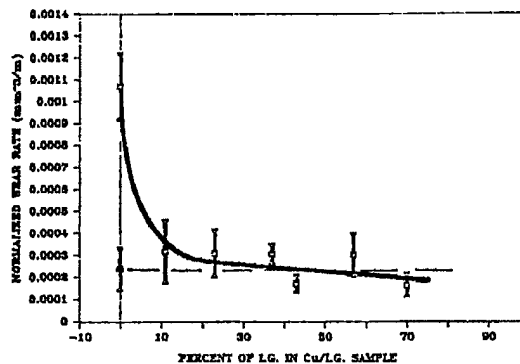


Fig. 8. Friction coefficient results for the Cu - IG composites. Dashed line represents baseline for 2-pin tests. Range of \pm std. dev. shown for each average value.



a



b

Fig. 9. (a) Wear rate results for Cu - IG composites. Dashed line represents wear rate baseline from 2-pin tests. (b) Same wear rate data normalized to volume (or area) fraction of Cu in the composite (in the wear scar area).

the wear volume per unit sliding distance per unit contact pressure for both the film (f) and the lubricant phase (lp) in the composite, can be expressed using the Archard-Holm wear equation as:

$$K_{lp} A_{lp} / H_{lp} \geq K_f A_f / H_f \quad (1)$$

where K is the wear coefficient, H the indentation hardness, and A the contact area of the lubricant phase and the interfacial film.

But $A_f = A_{\text{contact}}$; $H_{lp} = H_c(\text{composite})$; $K_{lp} = K_c(\text{composite})$

since the lubricant phase is part of the composite, as far as the load distribution in the contact region and wear are concerned.

$$\text{Then } K_c A_{lp} / H_c \geq K_f A_{\text{contact}} / H_f. \quad (2)$$

Thus, it is required for the wear coefficient of the composite if the interfacial film is to maintain a steady-state thickness:

$$K_c \geq K_f (H_c / H_f) (\text{lubricant fraction in composite})^{-1} \quad (3)$$

The observed decrease in wear rate (and thus wear coefficient) for the composite shown in Fig. 9 with increasing graphite fraction is consistent with Eqn. 3. This relation can be further refined to include composite micro-structural parameters such as phase diameter and mean-free-path.

5 CONCLUSIONS

Intercalated (NiCl_2) graphite in copper provides a lubricating, wear-reducing film when worn against 440C steel. Friction coefficients can be as low as 0.15 in air. Volume wear rate decreased by a factor of 5x with a full IG film present in the contact, and by a factor of 4x with a composite specimen containing 12 vol. % IG in copper. Increasing IG content above 15 vol. % leads to increasing wear due to loss of composite strength. Composite worn surface morphology shows three significant mechanisms: (i) lubricating interfacial film formation at the exit region of the soft phase, (ii) recession from the average surface at the soft phase locations, (iii) collection of wear debris in the recess at the entrance region, thus removing some of the wear debris but blocking part of the soft phase area.

6 ACKNOWLEDGEMENT

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REFERENCES

- (1) PETERSON, M.B. and RAMALINGAM, S., in Fundamentals of Friction and Wear of Materials, ed. D.A. Rigney (ASM, Metals Park, OH, 1981). pp. 331-372.
- (2) CONTE, A.A., ASLE Transactions **26**, 200 (1983).
- (3) LANCASTER, J.K. and MOORHOUSE, P., Tribology International **18**, 139-148 (1985).

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